
पेंट्स के लिए पानी में घुलनशील क्षारातु
कार्बोक्सीमिथाइल सेल्यूलोस — विशिष्टि

Water-Soluble Sodium
Carboxymethyl Cellulose
for Paints — Specification

ICS 87.040

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FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Raw materials for Paints, Varnishes and Related Products Sectional Committee had been approved by the Chemical Division Council.

This Indian Standard has been developed for sodium carboxymethyl cellulose used in paint industry. Sodium carboxymethyl cellulose, also known as cellulose gum and sodium cellulose glycolate, has a variety of uses in industries, such as paper, detergents, textiles, and oil drilling. It is also used in food, medicine, cosmetics, and paints. IS 3520 covers the requirements of different grades of the material for textile, detergent, insecticide, oil-well drilling and paper industries. IS 9830 covers the requirements for cosmetic industry and pharmaceutical industry. Food grade material is covered in IS 5306. The sectional committee felt that the requirements detailed in these standards are not adequately covering the sodium carboxymethyl cellulose used in paint industry. Therefore, the committee decided to formulate a separate standard to assist the manufacturers of Paints to procure material of requisite quality.

For the purpose of deciding whether a particular requirement of this standard is complied with the final value, observed or calculated, expressing the result of a test or analysis shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Indian Standard

WATER-SOLUBLE SODIUM CARBOXYMETHYL CELLULOSE FOR PAINTS — SPECIFICATION

1 SCOPE

This standard prescribes the requirements, and methods of sampling and test for water-soluble sodium carboxymethyl cellulose (Na-CMC) for use in paint industry.

2 REFERENCES

The following standards contain provisions, which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards.

<i>IS No.</i>	<i>Title</i>
1070 : 1992	Reagent grade water (third revision)
2316 : 1990	Method of preparation of standard solution for colorimetric and volumetric analysis (second revision)
2317 : 1975	Method for gravimetric determination of sulphates (first revision)
336 : 973	Specification of ether (second revision)
12305 : 2018	Laboratory sintered (fritted) filters, porosity grading; classification and designation (first revision)
101 (Part 8/Sec 5) : 1993	Methods of sampling and test for paints, varnishes, and related products: Part 8 Tests for pigments and other solids, Section 5 Lead restriction test

3 REQUIREMENTS

3.1 Colour and Appearance — The material shall be white to creamish powder. It shall be free from extraneous matter and other thixotropic materials, such as starches, gums or sodium alginate and shall be free-flowing, granular or fluffy.

3.2 Odour — The material shall be odourless.

3.3 Solubility in Water — The material shall readily disperse in water to give colloidal solution and shall give a clear solution on boiling.

3.4 Identification — The material shall not contain methyl or ethyl cellulose, reducing sugars, gelatine, and sodium alginate, and shall pass the following identification tests.

3.4.1 Distinction from Methyl Cellulose — Take 10 ml of 1 percent solution of the material. Adjust its pH to 0.5 with dilute hydrochloric acid. A fine white precipitate shall form.

3.4.2 Distinction from Methyl Cellulose and Ethyl Cellulose — Boil 10 ml of 1 percent aqueous solution of the material. No precipitate shall form.

3.4.3 Distinction from Reducing Sugars — To 5 ml of the 1 percent aqueous solution of the material, add 5 ml of dilute hydrochloric acid, boil under a reflux condenser for 20 min, cool and neutralize to litmus paper with sodium hydroxide solution; add 0.1 ml of this solution to 5 ml of hot potassium cupritartrate solution. No red precipitate shall be produced.

3.4.4 Distinction from Gelatine — To 10 ml of 1 percent aqueous solution, add 1 ml of copper sulphate solution, a blue precipitate shall form.

3.4.5 Distinction from Sodium Alginate — To 10 ml of 1 percent aqueous solution, add 10 ml of 1 percent (m/v) solution of calcium chloride in water. No gelatinous precipitate shall form.

3.5 Lead Restriction — The material shall be tested for restriction from lead in accordance with IS 101 (Part 8/Sec 5), or by Atomic Absorption spectrometer or by ICP. When thus tested the material shall not contain lead or compounds of lead or mixtures of both, calculated as metallic lead exceeding 90 ppm (see Note).

NOTE — When no lead is used during production, the product is considered lead free.

3.6 Toxic Heavy Metal Restriction — Product shall not be manufactured using mercury and mercury compounds, cadmium, chromium VI, arsenic, antimony,

and their oxides. The material shall not contain more than 0.1 percent by weight in total of above toxic heavy metals in the form of natural impurities or impurities entailed by the production process which are contained in the raw material when tested by the relevant atomic absorption spectroscopic methods.

3.7 The material shall also conform to the requirements given in Table 1.

4 PACKING AND MARKING

4.1 Packing

The material shall be securely packed in moisture-proof bags or rigid containers, as agreed to between the purchaser and the supplier. The containers shall be, such as to preclude contamination of the contents with metals or other impurities.

4.2 Marking

4.2.1 The packages and the drums or bags in which the material is stored shall be marked with the following:

- Name and description of the material;
- Name of the manufacturer and/or the trade-mark, if any;
- Net mass, and gross mass;
- Lead content (*Maximum*);
- Toxic heavy metals content;

f) Batch number in code or otherwise to enable the batch of manufacture to be traced back from records;

g) Month and year of manufacture;

h) A cautionary note as below:

- Keep out of reach of children; and
- This product may be harmful if swallowed or inhaled.

4.2.2 Certification Marking

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

6 TESTS

6.1 Tests shall be carried out as given Table 1 and described in relevant appendices to this standard.

6.2 Quality of Reagents

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be used in tests.

NOTE — Pure chemicals shall mean chemicals that do not contain impurities which affect the results of analysis.

8 SAMPLING

Representative samples of the material shall be drawn, as prescribed in IS 101 (Part 1/Sec 1).

Table 1 Requirements for Sodium Carboxymethyl Cellulose
(Clause 3.6)

Sl No. (1)	Characteristic (2)	Requirement (3)	Method of Test, Ref to Annex (4)
(i)	Active matter content, percent by mass (on dry basis), <i>Min</i>	92	A
(ii)	Loss on drying, percent by mass, <i>Max</i>	10	B
(iii)	Degree of substitution	0.55 to 1.0	C
(iv)	pH of 1 percent solution	6.0 to 8.5	D
(v)	Inorganic salts, free glycolates and other matter soluble in alcohol, percent by mass (on dry basis), <i>Max</i>	3	E
(vi)	Viscosity, Centipoise, <i>Min</i>	4000	F
(vii)	Viscosity stability, percentage retained vs. initial, <i>Min</i>	80	F

ANNEX A

[Table 1, Sl No. (i)]

DETERMINATION OF THE ACTIVE MATTER CONTENT

A-1 OUTLINE OF THE METHOD

Sodium carboxymethyl cellulose is precipitated from an aqueous solution by the addition of 95 percent ethanol. This is weighed and expressed as percentage of active matter content in the sample.

A-2 REAGENTS

A-2.1 Hydrochloric Acid — 1 N.

A-2.2 Sodium Hydroxide Solution — 1 N.

A-2.3 Ethyl Alcohol — 80 percent aqueous solution.

A-2.4 Ethyl Alcohol — 95 percent.

A-2.5 Ethyl Ether — (*see IS 336*).

A-2.6 Phenolphthalein Indicator Solution

A-3 PROCEDURE

Weigh about one gram of the material to the nearest 0.1 mg and place in a 250 ml beaker. Add 10 ml of hydrochloric acid and stir occasionally for 10 minutes. Add 20 ml of water and neutralize with sodium hydroxide using phenolphthalein as the indicator. The sample shall then be completely dissolved (check the solution to be neutral). Add about 0.2 ml of sodium hydroxide in excess. The sodium CMC is then precipitated by adding 160 ml of 95 percent alcohol under vigorous agitation. Add the alcohol first slowly

until a fine precipitate is formed, then add the rest. Allow the stirrer to run for about 15 min or until the precipitate settles rapidly and the supernatant liquid is clear. If a clear liquid is not obtained, place the beaker on a steam-bath for some time when the liquid becomes clear. Transfer quantitatively to an accurately weighed dry sintered glass filter crucible of porosity G 3 and filter by suction. Wash the beaker with 80 percent alcohol and transfer the washings to the crucible. Then wash the precipitate with 95 percent alcohol and finally, displace the alcohol by ether. Draw air through the crucible for about five minutes before placing it in a drying oven at 105 °C. Dry to constant mass (one to two hours) and weigh after cooling in a desiccator. Preserve the residue for test in C-1.2.

NOTE — If the sample contains sulphates, the dried active matter obtained in A-2.1 shall be ashed and the sulphate content determined gravimetrically following the procedure prescribed in IS 2317. The equivalent sodium sulphate content shall be subtracted from the final value.

A-4 CALCULATION

A-4.1 Active matter content,

$$\text{percent by mass} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the dry precipitate, and

M_2 = mass in g of the material taken for the test (on dry basis).

ANNEX B

[Table 1, Sl No. (ii)]

DETERMINATION OF LOSS ON DRYING

B-1 PROCEDURE

B-1.1 Weigh accurately about 5 g of the material in a weighed, clean, and dry squat form weighing bottle and dry to constant mass at 105 ± 2 °C. Cool in a desiccator and weigh.

B-1.2 Calculation

Loss on dry,

$$\text{percent by mass} = \frac{100 (M_1 - M_2)}{M_1 - M}$$

where

M_1 = mass in g of the weighing bottle with the material before heating,

M_2 = mass in g of the weighing bottle after heating, and

M = mass in g of the weighing bottle.

ANNEX C

[Table 1, Sl No. (iii)]

DETERMINATION OF DEGREE OF SUBSTITUTION

C-1 GENERAL

Two methods are described. Method 1 is the routine method. In case of dispute, method 2 shall be used as referee method.

C-2 METHOD A

C-2.1 Principle

The active ingredient recovered from the sample is incinerated to leave a residue of sodium carbonate which is determined acidimetrically. The amount of sodium carbonate, being directly related to the amount of sodium carboxymethyl groups in the material, is calculated in terms of the number of such groups per anhydroglucose unit and expressed as the degree of substitution.

C-2.1.1 Reagents

C-2.1.2 *Dilute Hydrochloric Acid* — Approximately 1 N.

C-2.1.3 *Sodium Hydroxide Solution* — Approximately 1 N.

C-2.1.4 *Ethyl Alcohol* — 95 percent.

C-2.1.5 *Ethyl Ether*

C-2.1.6 *Standard Sulphuric Acid* — 0.1 N (see IS 2316).

C-2.1.7 *Standard Silver Nitrate Solution* — 0.1 N (see IS 2316).

C-2.1.8 Methyl Red Indicator

C-2.2 Procedure

C-2.2.1 Take about 1 g material in a 250 ml beaker. Add 10 ml of dilute hydrochloric acid and stir occasionally for 10 min. Add 20 ml water and neutralize with 1 N sodium hydroxide solution using phenolphthalein as the indicator. The sample shall then be completely dissolved (check the solution to be neutral). Add about 0.2 ml of sodium hydroxide in excess. Then precipitate sodium CMC by adding 160 ml of 95 percent alcohol under vigorous stirring. Filter through a filter paper or filter crucible, weigh with 90 percent alcohol and displace the alcohol by ether. Dry in air and finally in an oven at about 105 °C.

C-2.2.2 Weigh about 0.5 g to the nearest 0.1 mg of the residue obtained in C-2.2.1 in a platinum crucible, ash very gently over a small flame or in a furnace. Dissolve the ash in water and titrate against 0.1 N sulphuric acid in a conical flask with methyl red as the indicator. When the first, not very sharp, end point is reached, heat to boiling so that the carbon dioxide is removed and then titrate to a new sharp end point. To ascertain that the ash does not contain sodium chloride, add potassium chromate, and titrate with silver nitrate. If more than three drops of silver nitrate are required, sodium chloride content of the isolated residue shall be determined and subtracted from the original mass of the residue. Similarly, if the ash contains sodium sulphate, it is to be determined separately and sodium sulphate content shall also be subtracted from the original mass of the residue (see IS 2317).

NOTE — The method shall not be used for samples containing phosphate.

C-2.3 Calculation

$$\text{Degree of substitution} = \frac{0.162 B}{1 - 0.08 B}$$

where

B = 0.1 b/C,

b = volume in ml of 0.1 N sulphuric acid required,
and

C = mass in g of residue taken.

C-3 METHOD B

C-3.1 Principle

This method involves conversion of sodium salt of the carboxymethyl cellulose to the acid form by treating with methanol acidified with hydrochloric acid or nitric acid, removal of the excess acid by washing with a methanol water solution and drying the material. Weighed sample of the free acid is dissolved in distilled water containing an excess of standard sodium hydroxide and the excess is back titrated with standard hydrochloric acid using phenolphthalein indicator.

C-3.2 Reagents

C-3.2.1 Dilute nitric acid — Add 100 ml of 70 percent nitric acid to 1 litre of methanol slowly with stirring.

C-3.2.2 Dilute hydrochloric acid — Add 25 ml of concentrated hydrochloric acid to a solution of 900 ml of methanol and 270 ml of distilled water.

C-3.2.3 Methanol — Anhydrous.

C-3.2.4 Methanol solutions — 70 percent (m/m) and 80 percent (m/m).

C-3.2.5 Standard hydrochloric acid — 0.5 N.

C-3.2.6 Standard hydroxide solution — 0.5 N.

C-3.3 Procedure

C-3.3.1 Place 10 to 15 g of freshly precipitated sodium carboxymethyl cellulose and 200 ml of dilute nitric acid in a 500-ml conical flask, stopper and shake for 3 to 4 h. Transfer the acid and CMC to the filter funnel and remove the acid liquor by suction. Add 100 to 150 ml of 70 percent methanol to the filter funnel, break up the mat by stirring, and then attach a 2-litre separating funnel to the top of the filter by means of a bored rubber stopper to give a tight seal. Place 2 litres of 70 percent methanol in the separating funnel and open the stopcock slightly to allow one drop per second to pass through the filter below. This washing may conveniently be carried on overnight.

NOTE — Use 80 percent methanol for washing if the degree of substitution is about 1.0.

C-3.3.2 When all the methanol has been delivered to the filter, place the filter on a filter flask and remove the excess solvent by suction. Add 100 to 150 ml of 70 percent methanol to the filter, stir up the CMC mat, and again remove excess methanol by suction. Test the filtrate for neutrality by mixing 5 ml of the filtrate with 5 ml of distilled water and adding a drop of methyl red indicator. If the filtrate is acidic, continue washing on the same manner until the filtrate is neutral to methyl red and then remove as much as possible of the wash liquor under strong suction.

C-3.3.3 Add 150 ml of anhydrous methanol to the filter, break up the CMC mat by stirring, stopper and allow to dehydrate for 1 hour. Again, remove the methanol under strong suction and then transfer the CMC to a small beaker. The remaining quantity of methanol may be removed by drying with a blower or in an oven at 80 °C.

C-3.3.4 Place approximately 2 g of the washed and dried CMC in a weighing bottle and dry for 1 hour at 100°C. Weigh by difference (to the nearest milligram) into a 100 ml conical flask with a rubber stopper. Add 15 ml of 70 percent methanol, allow to stand for few minutes and then add 20 ml of distilled water and 50 ml of 0.5 N sodium hydroxide solution, accurately measured from a burette. Place the flask on a bottle shaker and shake rapidly for 3 to 5 hours to dissolve or disperse the CMC. Titrate with standard hydrochloric acid using phenolphthalein indicator.

C-3.3.5 If dilute hydrochloric acid is used in **C-2.2.1** the sample of CMC shall be given three 1-hour steeps, using 100 ml of fresh reagent for each steep instead of the 3 to 4 h shaking period used with a single portion of nitric acid solution as described above. Either procedure may be used, although the nitric acid procedure is more convenient.

C-3.4 Calculation

Degree of substitution

$$(\text{DS}) \frac{0.162 S}{(1 - 0.058) S}$$

$$S \frac{Aa - Bb}{M}$$

where

A = volume in ml of standard sodium hydroxide solution used,

a = normality of sodium hydroxide solution,

B = volume in ml of standard hydrochloric acid used in **C-2.3.4**,

b = normality of hydrochloric acid, and

M = mass in g of sodium carboxymethyl cellulose taken for test in **C-2.3.4**.

ANNEX D[Table 1, *Sl No.* (iv)]**DETERMINATION OF pH****D-1 PROCEDURE**

D-1.1 Accurately weigh 1 g of the material (on dry basis) in a beaker and add to it 100 ml of

carbon dioxide-free water. Store for 16 h in carbon dioxide-free atmosphere. Determine the pH of this solution using a suitable pH meter.

ANNEX E[Table 1, *Sl No.* (v)]**DETERMINATION OF INORGANIC SALTS, FREE GLYCOLATES AND OTHER MATTER SOLUBLE IN ALCOHOL****E-1 PRINCIPLE**

A weighed sample is treated with 80 percent ethanol in which sodium CMC is insoluble, washed with 80 percent ethanol, dried and weighed. The difference in the weights is expressed as the alcohol-soluble matter.

E-2 PROCEDURE

To about 2 g of the sample, accurately weighed, add 250 ml of 80 percent ethanol and stir for 30 min. Transfer the residue to a tared sintered glass crucible with the aid of about 150 ml of ethanol (80 percent), followed by about 150 ml of ethanol (95 percent), wash

with ethyl ether until the alcohol is removed, draw air through the crucible until ether is removed and dry the residue to constant mass at 105 °C.

E-3 CALCULATION

Inorganic salts, free glycolates and other matter soluble in alcohol:

$$= \frac{M_2 - M_1}{M_2} \times 100$$

where

M_2 = mass in g, of the sample on dry basis; and

M_1 = mass in g, of the residue.

ANNEX F[Table 1, *Sl No.* (vi and vii)]**DETERMINATION OF VISCOSITY AND VISCOSITY STABILITY****F-1 PRINCIPLE**

F-1.1 The resistance to movement of a spindle is measured and expressed in terms of viscosity in seconds. The resistance being directly linked with viscosity can be expressed directly in terms of viscosity by previous calibration of the instrument.

F-2 APPARATUS**F-2.1 Brookfield Viscometer Type LVF or Equivalent****F-2.2 Mechanical Stirrer**

F-2.3 Constant Temperature Bath — maintained at 25 ± 1 °C.

F-3 PROCEDURE

F-3.1 Determine the moisture content of the sample. Calculate on dry basis the mass of sample necessary to make 1000 g test solution as follows:

$$\text{Mass of sample, g} = \frac{100 \times A}{100 \times B}$$

where

A = desired dry mass of sample in g, and

B = percentage of moisture in the sample as weighed.

F-3.2 Add 200 ml of distilled water to the jar, add sodium CMC and then add sufficient distilled water to

make a total of 1 000 g of solution. Place the stirrer in the solution so that the blade is about halfway between the bottom of the jar and the surface of the liquid, stir till the sample dissolves. Remove the agitator and transfer the sample container to the constant temperature bath and keep it there for 3 h. Remove the sample container from the bath, stir it again and divide it into 2 parts. Preserve 1 part of this solution in a suitable airtight glass bottle under nitrogen at ambient temperature for 6 days. Measure the viscosity of the first part with the Brookfield viscometer at 25 °C selecting the proper spindle and speed from Table 2. Allow the spindle to rotate until reading is obtained. Measure the viscosity of the second part after 6 days after keeping the sample container in the constant temperature bath for 3 hours.

NOTE — No preservative is added while preparing the solution, therefore, the container and water must be free of any microbial contamination.

**Table 2 Viscometer Spindle Required
for Given Speeds**

(Clause F-3.2)

Viscosity Range Cp	Spindle No.	Speed Rev/Min	Scale	Factor
(1)	(2)	(3)	(4)	(5)
10 to 100	1	60	100	1
100 to 200	1	30	100	2
200 to 1000	2	30	100	10
1000 to 4000	3	30	100	40
4000 to 10000	4	30	100	200

F-3 CALCULATION

F-3.1 Viscosity, initial, (Brookfield) in cP = Reading
× Factor

where

Reading = number obtained from the viscometer, and

Factor = number given in Table 2 for the spindle
and speed selected.

NOTE — Above calculation is not required for digital version of Brookfield viscometer as it directly displays the viscosity value.

F-3.1 Viscosity stability, percentage =

$$100 - \left[\frac{V_1 - V_2}{V_1} \times 100 \right]$$

where

V_1 = Initial viscosity in cP; and

V_2 = Viscosity after 6 days of stability in cP.

ANNEX K

(Foreword)

COMMITTEE COMPOSITION

Raw Materials for Paints, Varnishes and Related Products Sectional Committee, CHD 21

<i>Organization</i>	<i>Representative(s)</i>
In personal capacity (Retired from IIT Bombay)	PROF A. S. KHANNA (Chairman)
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Institute of Minerals and Materials Technology	NOMINATION AWAITED
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Amendments are issued to standards as the need arises on the basis of comments. Standards are also reviewed periodically; a standard along with amendments is reaffirmed when such review indicates that no changes are needed; if the review indicates that changes are needed, it is taken up for revision. Users of Indian Standards should ascertain that they are in possession of the latest amendments or edition by referring to the latest issue of 'BIS Catalogue' and 'Standards: Monthly Additions'.

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